NRL Memorandum Report 1457

FRACTOGRAPHY
PART XV. SOME ARTIFACTS POSSIBLE WITH THE TWO-STAGE PLASTIC CARBON REPLICATION TECHNIQUE

E. P. Dahlberg and C. D. Beachem

METALLURGY DIVISION

September 1963

U. S. NAVAL RESEARCH LABORATORY
Washington, D.C.

ABSTRACT

Several artifact structures are shown which are known to occur sometimes with the two-stage, plastic-carbon technique of fracture surface replication for electron microscopic observation. The origin and characteristic features of each artifact is discussed.

PROBLEM STATUS

This is an interim report; work on this problem is continuing.

AUTHORIZATION

NRL PROBLEM NUMBER: M01-08

BUREAU PROJECT NUMBER: RRMA 02 091/652-1/R007 06 01

INTRODUCTION

The two-stage plastic carbon replication technique is being widely used for electron microscope fractographic analysis of critical service failure items because it is relatively simple and leaves the fracture surface intact. By contrast, the direct carbon technique destroys the surface being replicated so that only a single replica can be made from any particular fracture area. The details of the two-stage fracture replication technique are described by Scott and Turkalo (1). Briefly, in the two-stage technique preliminary plastic replicas are moulded on the fracture surface itself. These replicas are shadowed with an evaporated film of heavy metal (e.g., palladium) and uniformly coated with a supporting layer of carbon. The plastic is dissolved away leaving a thin shadowed carbon film which reproduces the surface configuration of the fracture.

Replicas prepared by this technique often reveal artificial structures unrelated to the actual fracture surface topography. These artifacts, in many instances, can confuse the fractographic interpretation for the novice. The purpose of this report is to illustrate several of these known artifacts and discuss their origins and characteristic features.

ARTIFACTS RESULTING FROM UNCLEAN FRACTURES

If the maximum information is to be obtained from electron microscope observations the fractures must be replicated in a "clean" condition. Since the details of interest are of a very fine scale they can easily be obliterated by "after the fact" corrosion and handling. The presence of any dirt, rust, or other foreign matter on the fracture surface will greatly hamper the microscopic observations. Figure 1 shows the characteristic feature associated with the presence of rust on fractures of ferrous materials. The several large black areas are portions of the rust deposit which were lifted off the fracture surface by the initial plastic replica. The surface structure of the rust deposit which remained attached to the fracture is exhibited in the surrounding regions. The layer of rust appears to break into several small fragments. Replicas of the surface cracks around each of these small fragments produces the polygonized structure evident in the fractograph.

Figure 2 shows an artifact which resulted when a liquid rust preventative (immiscible in acetone) was not completely removed from a fracture prior to replication. The bubble-like structure resulted from replicating the liquid which remained on the fracture surface.

ARTIFACTS RESULTING FROM THE PLASTIC REPLICATION MATERIAL AND TECHNIQUES

The most widely used plastic material for two-stage replication of fractures is cellulose acetate. If the cellulose acetate, which is softened in acetone and moulded against the fracture, is removed before it has hardened sufficiently, it will pull away from the fracture unevenly and greatly distort the replica. The numerous finger-like protrusions shown in Figure 3 resulted from premature stripping of soft cellulose acetate.

In several instances the use of a highly concentrated solution of parlodian in amyl-acetate for primary replicas resulted in artifacts of the type shown in Figure 4. The bubble-like artifact is thought to be associated with moisture which dissolves in the thick replication liquid. Generally the bubbles develop randomly over the entire surface but, in some cases, they concentrate along particular features of the fracture such as fatigue striations or cleavage tear lines. The bubbles develop in the liquid replicating solution just above the fracture surface, leaving a thin plastic layer between the metal and the bubble. Figure 5 shows an area in which this thin film has been partially or completely torn away.

In stripping plastic replicas from fractures which contain highly irregular surface contours it is possible to tear small segments of plastic from the main replica. The torn plastic surface can then become part of the surface observed in the electron microscope. Figure 6 shows the characteristic appearance of this type of plastic tear.

Another artifact which may be produced during the plastic stripping operation is shown on the sides of rupture dimples in Figure 7. The parallel lines are formed when part of the plastic replica scrapes against the fracture surface during the stripping operation. The lines, which are generally uniformly spaced are observed on sides of hills or on slopes between elevation differences in the replica. The particular geometry necessary for scraping the plastic against the fracture is satisfied for these configurations. Figure 8 shows a precisely matched area on both halves of a fracture surface. The scraping artifact appeared on only one-half of the surface. This artifact is of special significance since it so closely resembles the characteristic striation topography of fatigue striations.

Figure 9 shows the appearance of undissolved plastic which has not been completely removed from the final carbon replica.

ARTIFACTS ASSOCIATED WITH THE CARBON REPLICA

The final carbon replica is prone to tears and folds since it is very thin and fragile. The number of artifacts of this type, illustrated in Figure 10, can be reduced considerably by careful preparation and handling techniques. The collapse of carbon replicas on areas with abrupt changes in elevation cannot, however, be avoided by techniques available today. Figure 11 shows areas where the carbon replica has collapsed in regions between cleavage facets on a fracture surface of mild steel.

Overheating shadowed carbon replicas during observation in the electron microscope can cause the heavy metal shadowing material to coalesce. Figure 12 shows the granular appearance of this artifact.

CONCLUSIONS

Artifacts introduced during the preparation, handling, and observation of two-stage plastic replicas can confuse and obscure fractographic analysis unless they are clearly recognized and understood. Extreme caution must be taken when interpreting particular replica structures which are either newly or infrequently observed.

ACKNOWLEDGEMENT

The authors gratefully acknowledge Drs. B. F. Brown, R. A. Meussner, and Mr. A. J. Edwards for their valuable assistance in analysing and interpreting the artifact structures shown in this report.

REFERENCE

1. R. L. Scott and A. M. Turkalo, Proc ASTM, Vol. 57, 1957, p. 536.

Fig. 1 - Characteristic appearance of a rust formation on a fracture of mild steel

Fig. 2 - Improperly cleaned fracture surface showing drops of liquid which was not removed prior to replication

Fig. 3 - Artifact structure which developed when a plastic replica was removed from a polished and etched surface before drying completely

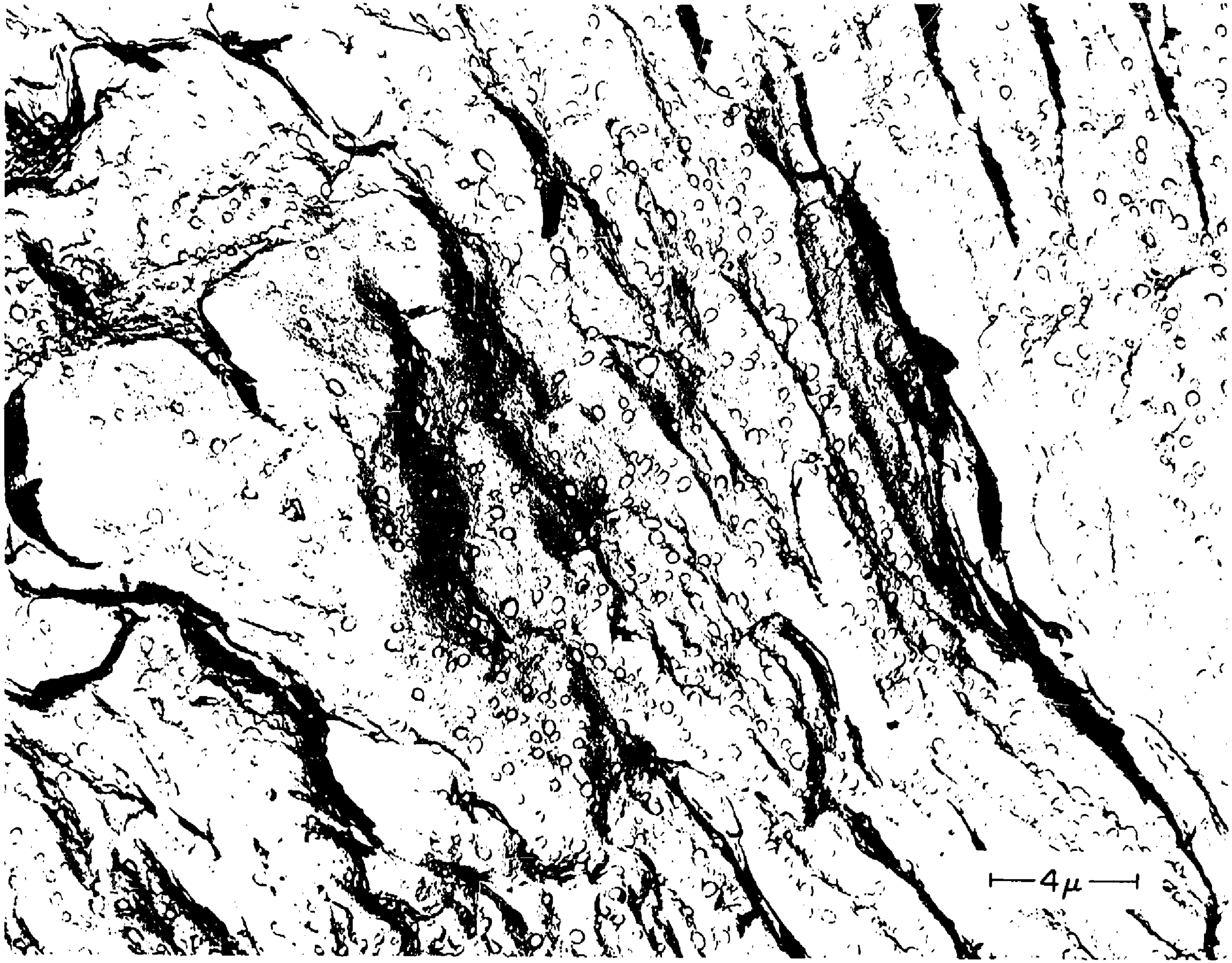

Fig. 4 - Bubble artifacts observed on a fatigue fracture replicated with a 10% solution of parlodion in amyl acetate

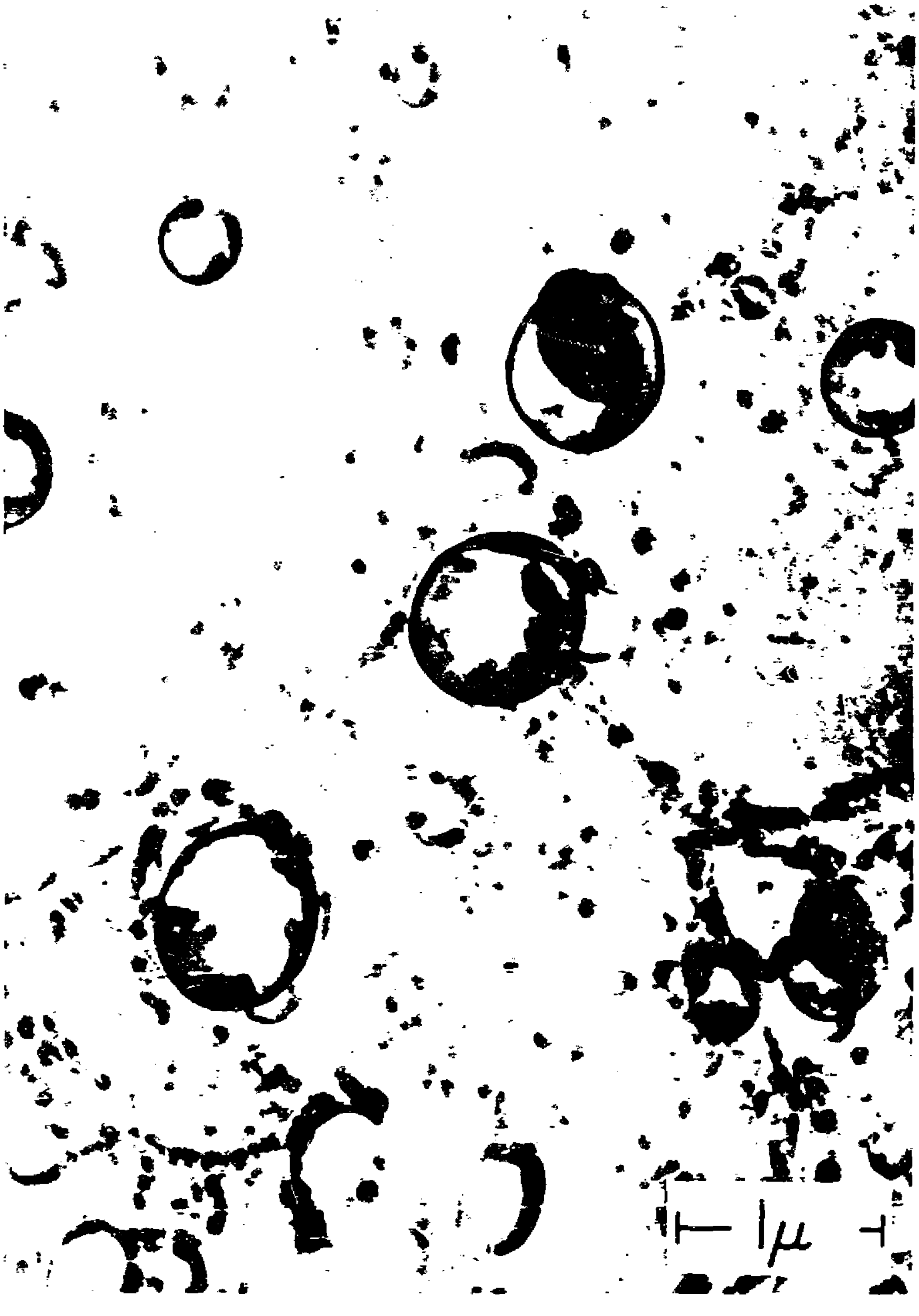

Fig. 5 - Enlarged bubble artifact showing partial and complete tearing of the plastic film which separated the bubble from the metal surface

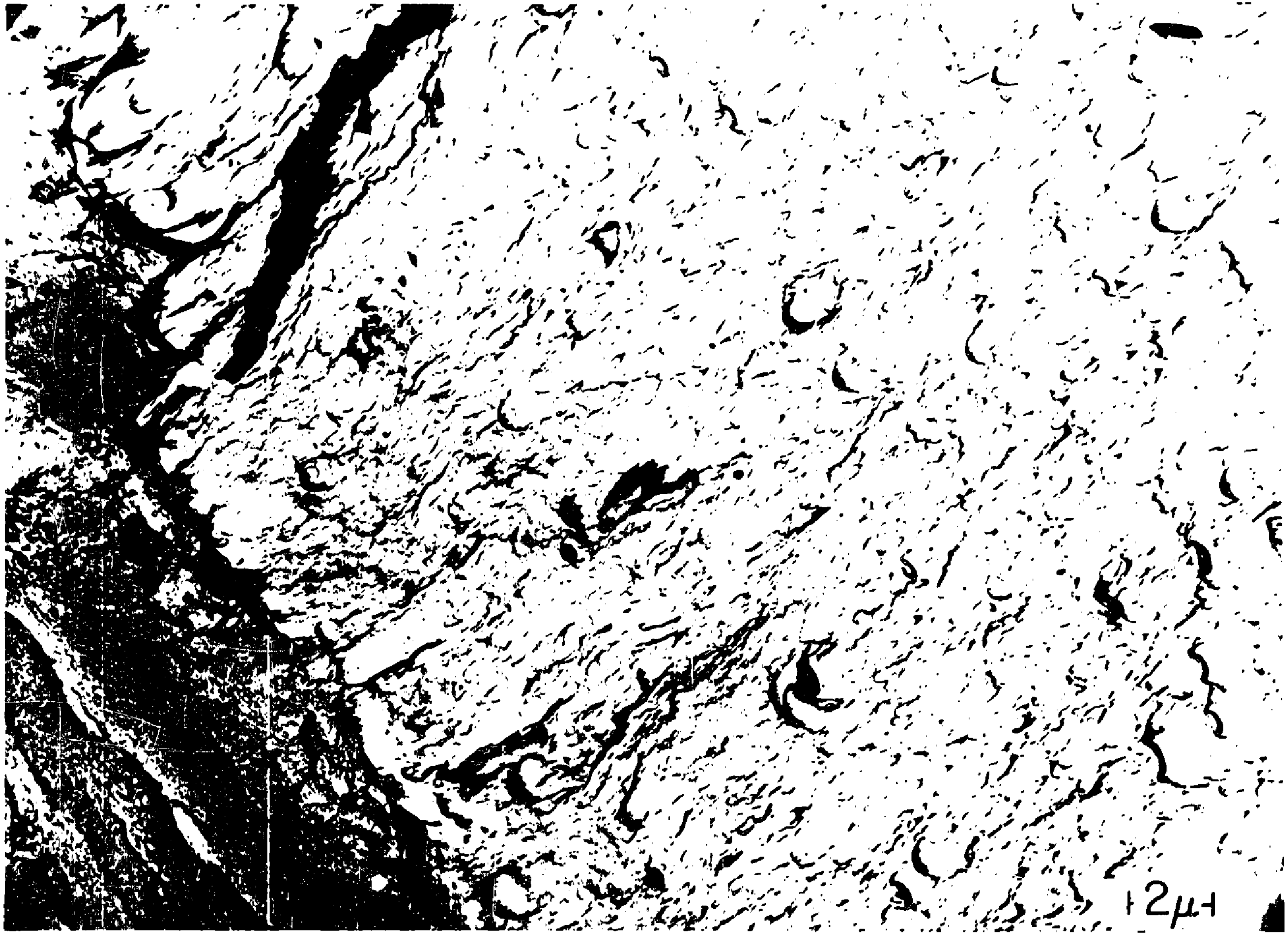

Fig. 6 - Surface structure of a tear in plastic of the first-stage replica

Fig. 7 - Artifact structure developed on the sides of rupture dimples due to scraping plastic replica against fracture face

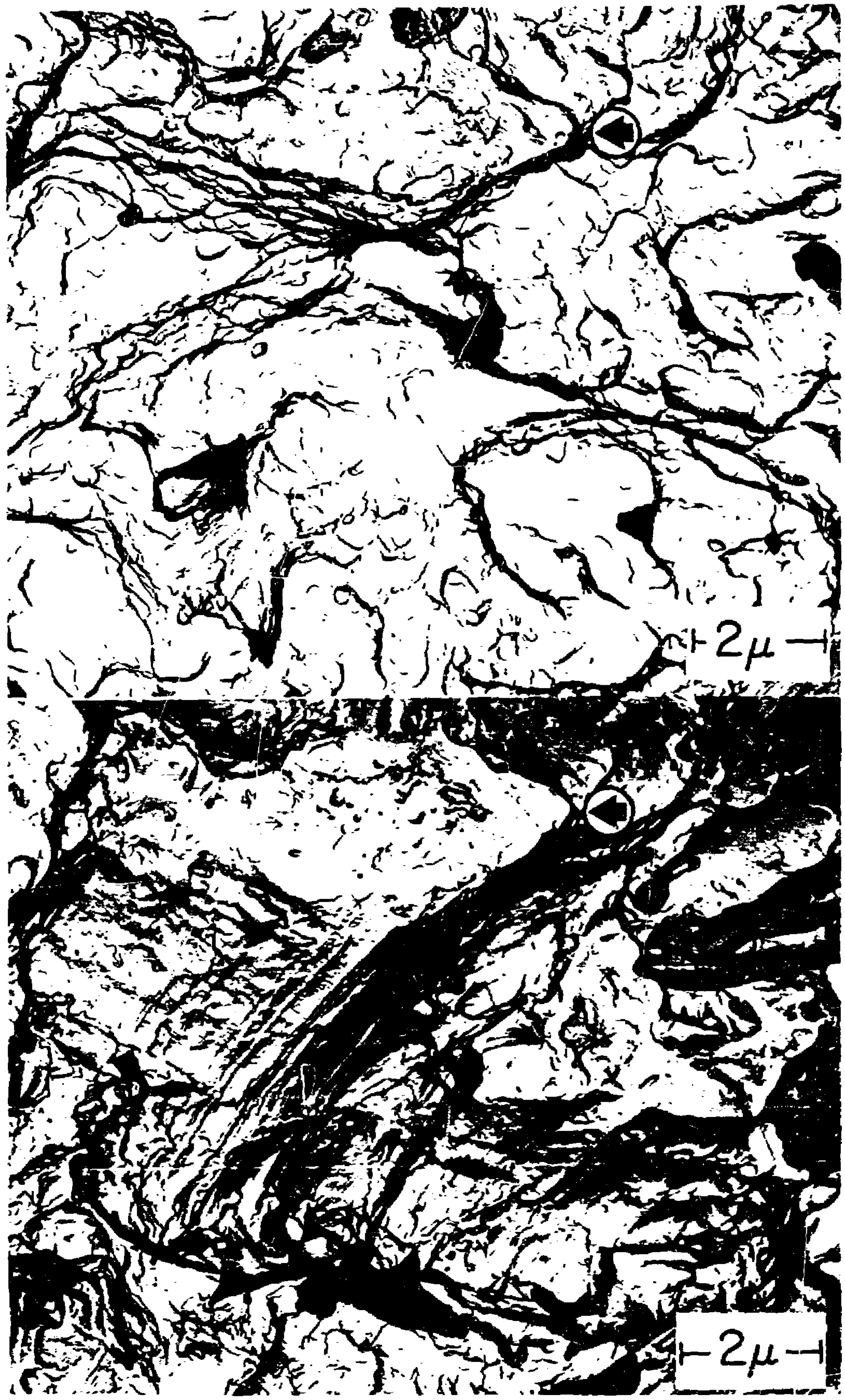

Fig. 8 - Precisely matched area on both halves of a fracture specimen showing presence of "scraping" artifact on only one piece. (Arrows point to one set of matching features.)

Fig. 9 - Undissolved plastic on final carbon replica

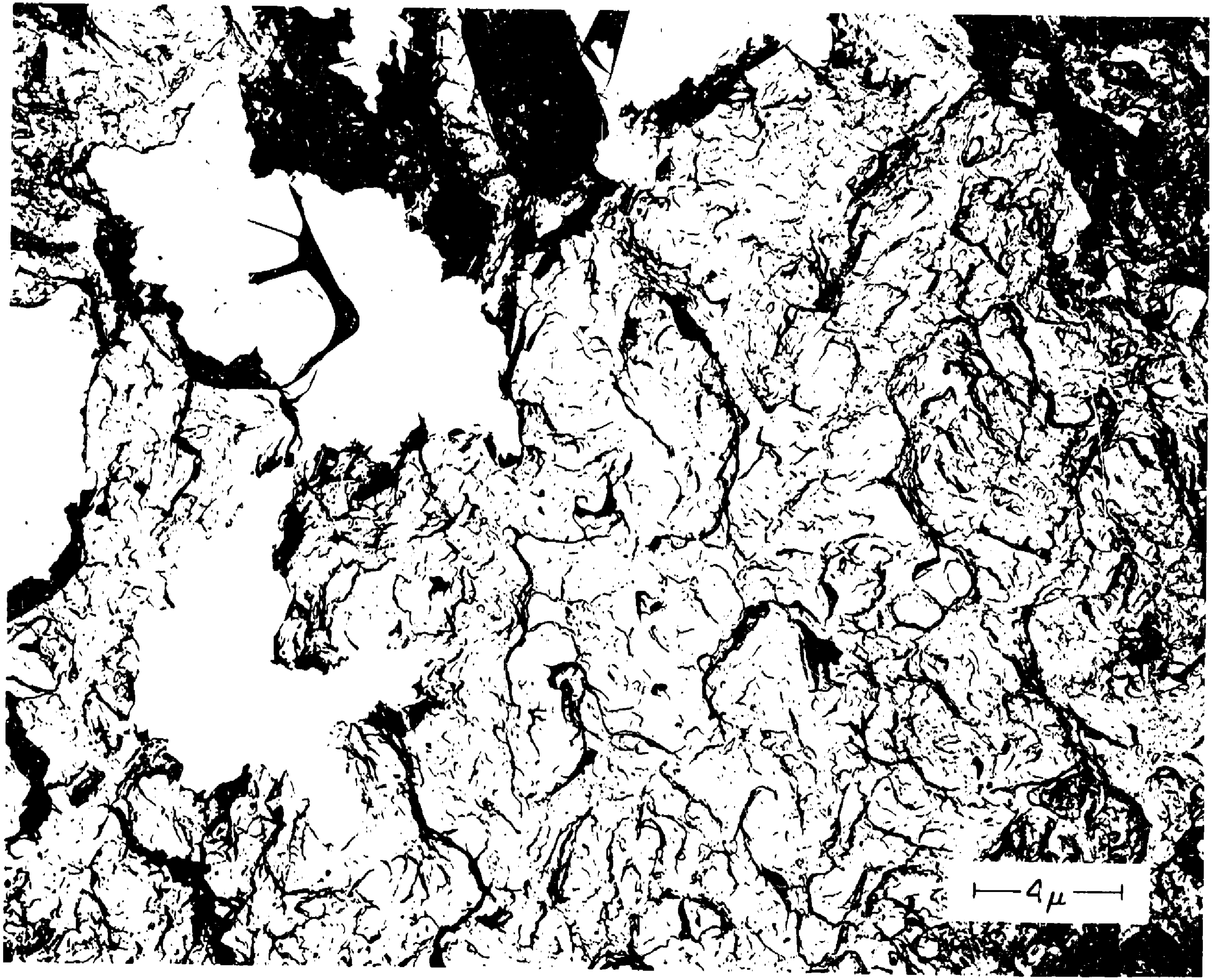

Fig. 10 - Folds and tears in final carbon replica

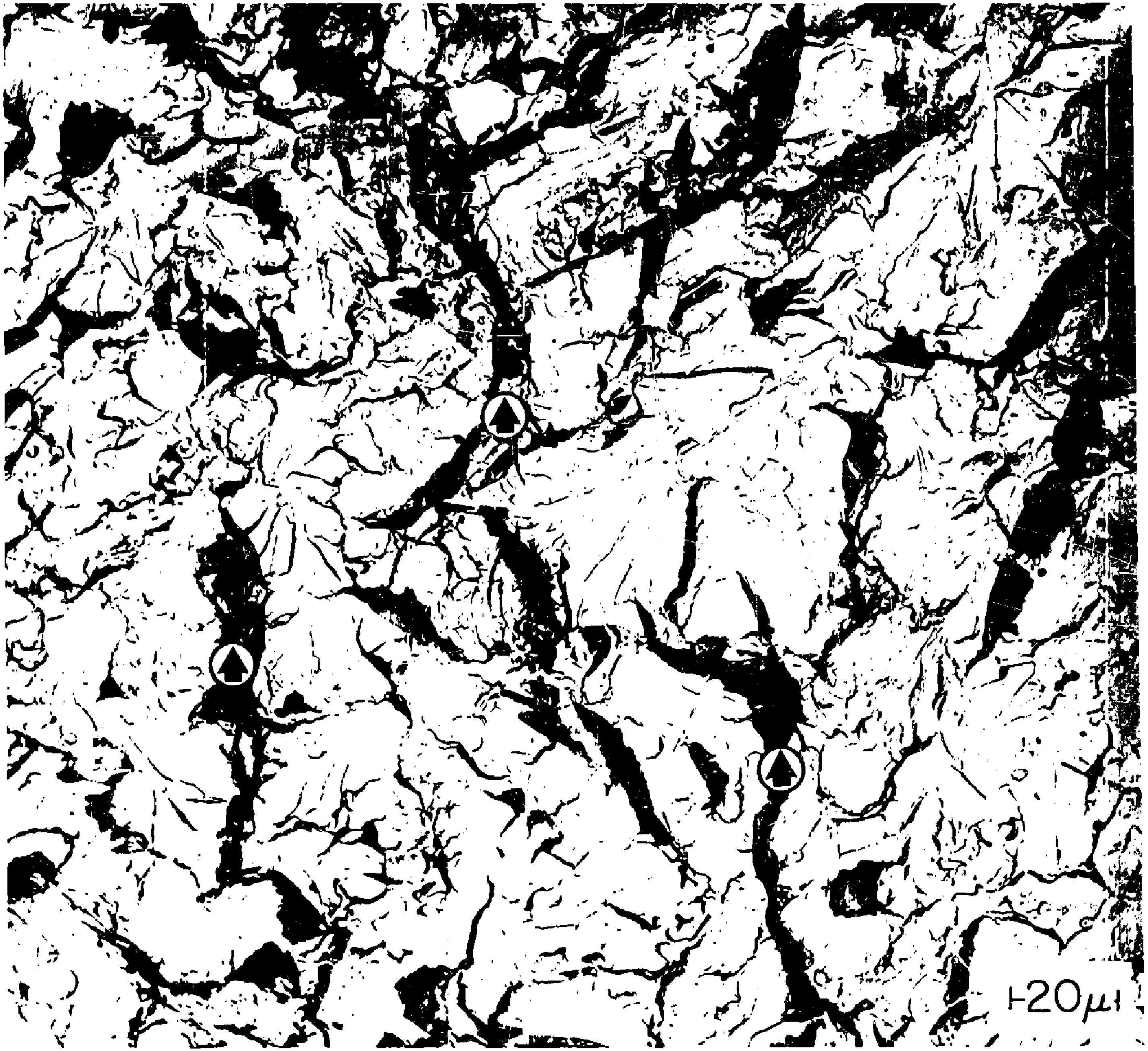

Fig. 11 - Collapsed carbon films (arrows) on replica of cleavage fracture in mild steel

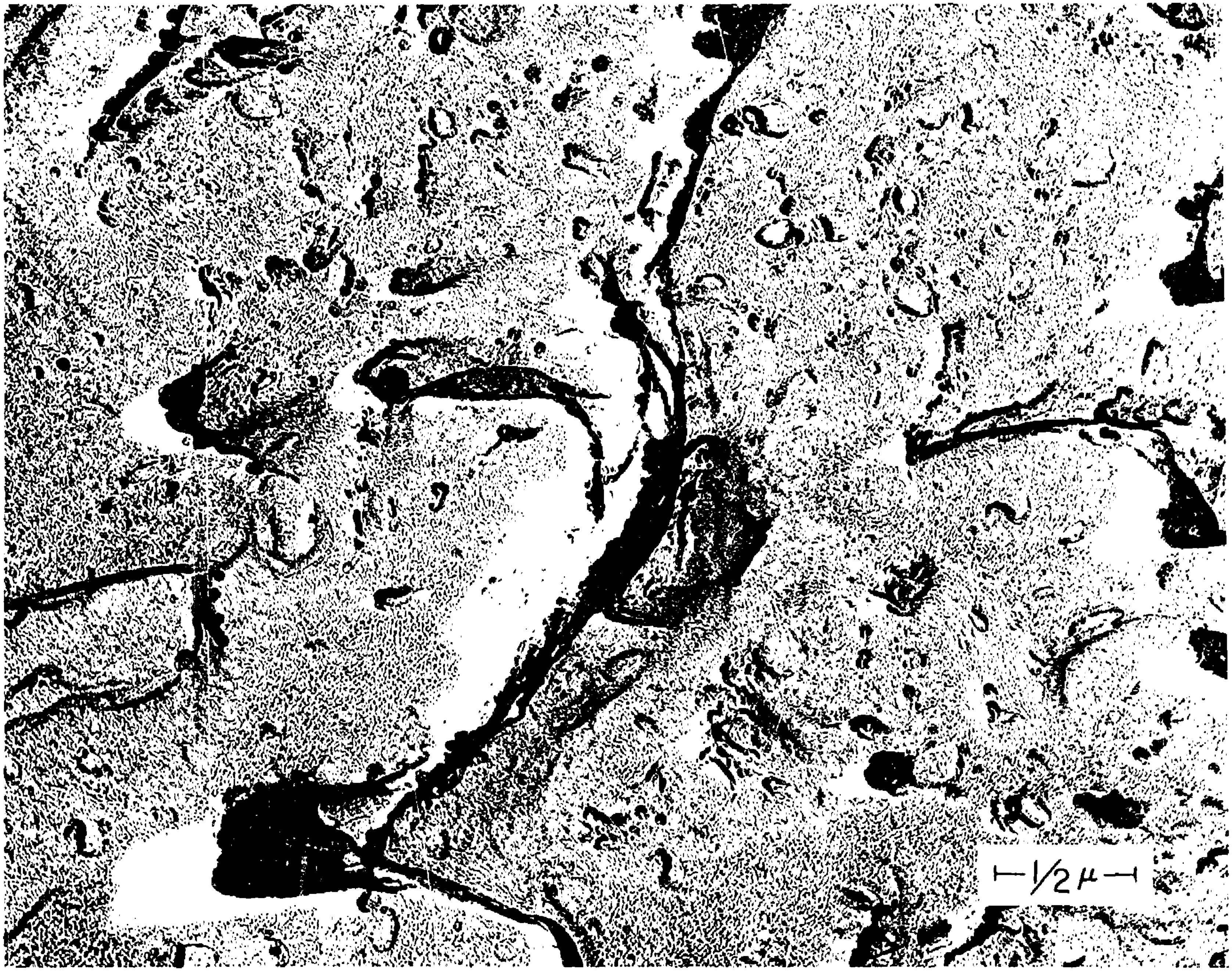

Fig. 12 - Granulated palladium shadowing material caused by overheating carbon replica in electron beam